CHEMISTRY PRACTICALS LIST AND PROCEDURE

List of Practicals to be done in the serial order

Preparation of inorganic compound

1. Preparation of Potash alum {$K_2SO_4.Al_2(SO_4)_3.24H_2O$}

Preparation of organic compound

2. Preparation of Acetanilide {$C_6H_5(NH)COCH_3$}

Titrymetry :-

a. Acid-Base titrations:

3. NaOH Vs HCl (Acidimetry)

b. Redox titrations:

4. Oxalic acid Vs $KMnO_4$ (Permanganometry)

5. Mohr's salt Vs $KMnO_4$ (Permanganometry)

Chemical kinetics

6. Saponification of ethyl acetate with NaOH

Qualitative analysis :

7. Semi micro analysis of **inorganic salts**

8. Identification of functional group of **organic compounds**

CHEMISTRY PRACTICALS LIST AND PROCEDURE

PROCEDURE :-

PREPARATION OF INORGANIC COMPOUNDS

1.1 Preparation of potash alum

Aim:

To prepare a pure sample of potash alum. **[$K_2SO_4.Al_2(SO_4)_3.24H_2O$]**

Principle:

Potash alum is prepared by dissolving an equimolar mixture of aluminium sulphate and potassium sulphate in minimum amount of water containing a little amount of sulphuric acid and then subjecting the solution to crystallization; octahedral crystals of potash alum separate out.

$$K_2SO_4 + Al_2(SO_4)_3 + 24H_2O \longrightarrow \underset{\text{(Potash alum)}}{K_2SO_4.\,Al_2(SO_4)_3.\,24H_2O \text{ [or] } 2KAl(SO_4)_2.\,12H_2O}$$

Apparatus required:

- Beaker (100 mL)
- Trough
- Funnel
- Wire gauze
- Conical flask (100mL)
- Glass rod
- Tripod stand

Chemicals required:

- Aluminium sulphate : 10 g
- Water : 20 mL
- Potassium sulphate : 2.5 g
- Dilute Sulphuric acid : 1 mL

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Procedure:

a. Take 20 mL of distilled water in a 100 mL beaker and heat it to about 40°c. Add about 1 mL of dilute sulphuric acid, warm this and dissolve 10 g of aluminium sulphate adding small amounts at a time.
b. Weigh 2.5 g of powdered potassium sulphate (given in the packet) and add it to the above solution, stir well.
c. Heat the solution with constant stirring till potassium sulphate dissolves completely on a water bath. Test the solution at regular intervals of time for crystallization point.
d. Allow the solution to cool to room temperature.
e. On cooling, white crystals of potash alum separate out.
f. Decant the mother liquor carefully and shake gently with a mixture of (1 cc alcohol + 1 cc water) to the crystals mixture.
g. Filter the crystals, dry these between the folds of a filter paper and note the yield.

Results:

Weight of crystals obtained = ………………… g

Colour of the crystals = ………………….

The crystals of potash alum are octahedral in shape.

Precautions:

a. Cool the solution slowly to get good crystals. Avoid rapid cooling.
b. Do not disturb the solution while cooling.

Answer the following question:

1. Why do we take equimolar quantities of reacting compounds in the preparation of Alum?

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Procedure for Preparation of Acetanilide

Aim:

To prepare a sample of Acetanilide from Aniline.

Theory:

Acetanilide is prepared by acetylating aniline with acetic anhydride in presence of glacial acetic acid. The chemical equation can be written as.

$C_6H_5NH_2 + (CH_3CO)_2O \rightarrow C_6H_5NHCOCH_3 + CH_3COOH$

Apparatus:

Conical flask (100ml), Water condenser, Wire-gauze, Tripod stand, Burner, Iron-stand, Clamp, measuring cylinder, etc.

Chemicals Required:

1. Aniline = 10 ml
2. Acetic anhydride = 10 ml
3. Glacial acetic acid = 10 ml

Procedure:

1. Take 10 ml of acetic anhydride in a clean dry 250 ml conical flask and add 10 ml of glacial acetic acid and shake the contents thoroughly.
2. To this mixture taken in a flask add 10ml of aniline and fit a water condenser.
3. Place the flask on a wire-gauze placed on a tripod stand.
4. Boil the mixture for 10 minutes.
5. Detach the condenser and pour the liquid into about 200ml ice cold water contained in a beaker. During addition, stir vigorously the contents of the beaker with the help of glass rod.
6. Filter the white precipitates which separate out and wash with coldwater.

7. *Crystallization of acetanilide*: Take the crude acetanilide in a beaker and add about 50ml of a mixture of 1 volume of acetic acid and 2 volumes of water. Heat to dissolve the solid. Filter the hot solution into a conical flask and cool the filtrate in ice. Filter off the crystals and dry them by pressing them between folds of filter paper. Weigh the crystals and record its yield.

Result:

Weight of acetanilide obtained , = ------- g (m.p. 113^{o}c)

Precautions:

1. Freshly distilled aniline should be used in order to get good results or small amount of Zinc can be added in the reaction mixture. Zinc reduces the colored impurities in the aniline and also prevents its oxidation during the reaction.
2. Prolonged heating and use of excess of acetic anhydride should be avoided.
3. Reaction mixture should first be cooled and then poured in ice-cold water otherwise hydrolysis of acetanilide may takes place.

Volumetric analysis – I

NaOH vs. HCl

Aim :- To determine the amount of NaOH in the given solution.

Requirement:- 0.1M HCl

Chemical equation:- $NaOH + HCl \longrightarrow NaCl + H_2O$

Indicator:- Phenolphthalein.

End point:- pink to colourless.

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Apparatus used:-

Burette. 25ml

Pipette 10ml

Titration flask.

Standard flask.

Procedure:-

- Burette is rinsed first with given HCl solution and then filled up to mark.
- First given NaOH is standardized by using standard flask with the help of water up to the mark.
- Pipette is taken and rinsed with the standard NaOH solution and pipette out 10ml of the solution in to the titration flask.
- 1 – 2 drops of phenolphthalein indicator are added to the titration flask. The solution becomes pink in colour.
- The flask is placed on the glazed tile under the burette and the initial reading in burette is noted.
- The acid solution in the burette is released slowly and drop wise with constant shaking until the end point is reached i.e. the pink colour is just discharged.
- At the end point pink colour of the solution disappears.
- The final reading in the burette is noted.
- Wash the titration flask with ordinary water and finally with distilled water.
- The procedure is repeated 4 – 5 times to get a set of at least three concurrent readings.
- The readings are tabulated in the following table.

Observations:-

S.No.	Initial reading of the burette	Final reading of the burette	Volume of acid used.
1	–	–	– ml
2	–	–	– ml

3	–	–	– ml
4	–	–	– ml

Calculations:-

HCl solution

Molarity of given HCl, M_1 = 0.1 M

Volume of HCl consumed, V_1= --ml

Number of moles of HCl ,n_1 = 1

NaOH solution

Volume of NaOH taken, V_2ml = 10ml

Molarity of NaOH taken = M_2 = ?

Number of moles of NaOH , n_2 = 1

Calculation of molarity of NaOH solution:-

Formula : $$\frac{M1V1}{n1} = \frac{M2V2}{n2}$$

$$M2 = \frac{M1V1}{n1} X \frac{n2}{V2}$$

By substituting all the known values , $M2 = \frac{0.1V1}{1} X \frac{1}{10}$

Therefore, $M2 = \frac{V1}{100}$

Calculation of strength of NaOH in g/litre:-

Strength per litre = molarity x molecular mass.

$$= \frac{V_1}{100} \times 40 \ (\because \text{molecular mass of NaOH} = 40)$$

$$= \frac{V_1}{5} \text{g / litre}$$

Questions:

1. **What is indicator?**

Solution:

Indicator is a chemical substance which changes colour at the end point.

2. **What is end point?**

   Solution:

   The stage during titration at which the reaction is just complete is known as the end point of titration.

3. **Why a titration flask should not be rinsed with the reacting solution?**

   Solution:

   This is because during rinsing some liquid will remain sticking to the titration flask, therefore the pipette volume taken in the titration flask increases.

4. **Burette and pipette must be rinsed with the solution with which they are filled, why?**

   Solution:

   The burette and pipette are rinsed with the solution with which they are filled in order to remove any substance sticking to their sides, which otherwise would decrease the volume of the liquids to be taken in them.

Volumetric analysis – 2

$KMnO_4$ vs $(COOH)_2$

Aim:

To determine the amount of $KMnO_4$ in the given solution

Requirement:

0.02 M oxalic acid

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Chemical equation:

$$2KMnO4 + 3H2SO4 + 5H2C2O4 \rightleftharpoons K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$$

Indicator:

$KMnO_4$ is self – indicator

End point:

Colourless to permanent pink colour

Apparatus used:

Burette – 25 ml

Pipette – 10 ml

Titration flask

Standard flask

Procedure:

- Burette is rinsed first with given potassium permanganate solution and then filled upto the end mark.
- Give oxalic acid standardized in standard flask upto the mark.
- Pipette is rinsed with the oxalic acid first, and then 10 ml of oxalic acid is pipette out into a titration flask.
- 10 ml of dilute sulphuric acid is added to the solution in the titration flask.
- Initial reading of the burette is noted, and the flask is heated to 60 – 70^0 c and then $KMnO_4$solution in the burette is added slowly and drop wise with constant shaking until a permanent light pink colour is imparted to the solution in the titration flask.
- Final reading in the burette is noted and the process is repeated 4 – 5 times to get a set of at least three concurrent readings.
- The readings are tabulated in the following table.

Observations:

S.NO	Initial reading of the burette	Final reading of the burette	Volume of $KMnO_4$ used
1.	-	-	- ml
2.	-	-	- ml
3.	-	-	- ml
4.	-	-	- ml

Calculations:-

$KMnO_4$ solution

Molarity of given $KMnO_4$, M_1 = ?

Volume of $KMnO_4$ consumed, V_1= --ml

Number of moles of $KMnO_4$,n_1 = 2

Oxalic acid solution

Molarity of $(COOH)_2$, M_2 = 0.02M

Volume of $(COOH)_2$ taken ,V_2ml =10ml

Number of moles of$(COOH)_2$, n_2 = 5

Calculation of molarity of $KMnO_4$ solution:-

Formula : $$\frac{M1V1}{n1} = \frac{M2V2}{n2}$$

$$M1 = \frac{M2V2}{n2} X \frac{n1}{V1}$$

By substituting all the known values , $M1 = \frac{0.02X10}{5} X \frac{2}{V1}$

Therefore, $M1 = \frac{2}{5} X \frac{0.2}{V1}$

Calculation of strength of $KMnO_4$ in g/litre:-

Strength per litre = molarity x molecular mass.

$$= \frac{2}{5} \times \frac{0.2}{V_1} \text{ x } 158 \ (\because \text{molecular mass of } KMnO_4 = 158)$$

$$= \frac{4}{50V_1} \times 158 \text{ g / litre}$$

Volumetric analysis – 3

$KMnO_4$ vs Ferrous ammonium sulphate

Aim:

To determine the atomic mass of the alkali metal. The given solution has been prepared by dissolving 1.6 g of an alkali metal permanganate($AMnO_4$) per litre of solution.

Requirement:

0.05 M ferrous ammonium sulphate solution

Chemical equation:

$$MnO_4^- + 8H^+ + 5Fe^{2+} \longrightarrow 5Fe^{3+} + Mn^{+2} + 4H_2O$$

Indicator:

$AMnO_4$ will acts as self – indicator

End point:

Colourless to permanent pink colour

Apparatus used:

Burette – 25 ml

Pipette – 10 ml

Titration flask

Standard flask

Procedure:

- Burette is rinsed first with given potassium permanganate solution and then filled upto the end mark.
- Give ferrous ammonium sulphate (Mohr's salt) is standardized in standard flask upto the mark.
- Pipette is rinsed with the ferrous ammonium sulphate first, and then 10 ml of oxalic acid is pipette out into a titration flask.
- 10 ml of dilute sulphuric acid is added to the solution in the titration flask.
- Initial reading of the burette is noted, and then $AMnO_4$ in the burette is released slowly and drop wise into the titration flask till a a permanent light pink colour is just imparted to the solution in the titration flask.
- Final reading in the burette is noted and the process is repeated 4 – 5 times to get a set of at least three concurrent readings.
- The readings are tabulated in the following table.

Observations:

S.NO	Initial reading of the burette	Final reading of the burette	Volume of $AMnO_4$ used
1.	-	-	- ml
2.	-	-	- ml
3.	-	-	- ml
4.	-	-	- ml

Calculations:-

$AMnO_4$ solution

Molarity of given $AMnO_4$, M_1 = ?

Volume of $AMnO_4$, V_1= --ml

Number of moles of $AMnO_4$,n_1 = 1

Mohr's salt solution

Molarity ofMohr's salt solution, M_2 = 0.02M

Volume of Mohr's salt solution,V_2ml =10ml

Number of moles ofMohr's salt , n_2 = 5

Calculation of molarity of $AMnO_4$ solution:-

Formula : $$\frac{M1V1}{n1} = \frac{M2V2}{n2}$$

$$M1 = \frac{M2V2}{n2} X \frac{n1}{V1}$$

By substituting all the known values , $M1 = \frac{0.05X10}{5} X \frac{1}{V1}$

Therefore, $$M1 = \frac{0.5}{5} X \frac{1}{V1}$$

Strength of $AMnO_4$ per litre = 1.6 g/litre.

Molecualar mass of alkali metal permanganate $= \frac{strength}{molarity}$

$$= \frac{1.6}{\frac{5}{50\,V}} = 16\ V_1$$

But molecular mass of AMnO4 = atomic mass of A + foumula mass of MnO_4^-

$$= M_A + 119$$

There fore from above observations $16\ V_1 = M_A + 119$

$$M_A = 16\ V_1 - 119$$

DETERMETINATION OF ORDER OF SAPONIFICATION OF ETHYL ACETATE BY NaOH

Aim:

To determine the order of saponification of ethyl acetate by NaOH from its kinetic study.

Apparatus:

Conical flasks – 2, burette 50 ml – 1, pipette 10 ml – 1, measuring jar.

Chemicals:

$\frac{M}{40}$ Ethyl acetate, $\frac{M}{40}$ NaOH, and $\frac{M}{20}$ HCl, Solution, $\frac{M}{50}$ NaOH.

Principal:

The reaction of saponification of ethyl acetate by NaOH may be represented as

$$CH_3COOC_2H_5 + NaOH \rightarrow CH_3COONa + C_2H_5OH$$

The rate law for the above equation is as follows:

$$\frac{dx}{dt} = k_2\,(CH_3COOC_2H_5)\,(NaOH)$$

As the reaction proceeds, NaOH is consumed; hence the progress of the reaction may be studied by finding the confrontation of HCl by titrating with standard $\frac{M}{50}$ NaOH at different intervals of time.

Procedure:

Take 50 ml of M/40 ethyl acetate solution into a stopper bottle and add 50 ml of M/40 NaOH to it, while adding note the time and mix it. Immediately pipette out 10 ml of reaction mixture into a conical flask already containing 20 ml of ice cold M/20 HCl solution. Titrate with M/50 NaOH using phenolphthalein indicator till light permanent pink colour pink colour appears. Note the burette reading i.e. V_0. Repeat the procedure for every 10 minutes interval of time, (V_t). Tabulate the readings in the form given.

Reaction mixture:

$$\left[50\ \text{ml}\ \frac{M}{40}\ CH_3COOC_2H_5 + 50\ \text{ml}\ \frac{M}{40}\ NaOH\right]$$

Indicator: Phenolphthalein;

End point: Colourless to pink.

		Burett readings	

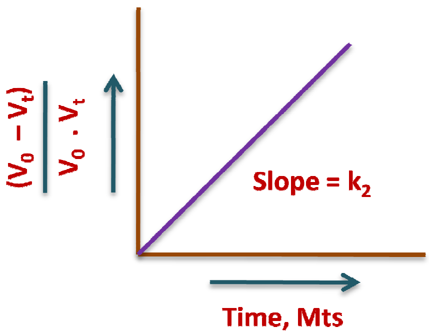
S.No	Time ‘t’ in min	V_0 intial volume	V_t Final vaolume	$(V_0 - V_t)$	
1	0	0			
2	10				
3	20				
4	30				
5	40				
6	∞				

$a \propto V_0$, $(a - x) \propto V_t$, $x = (V_0 - V_t)$

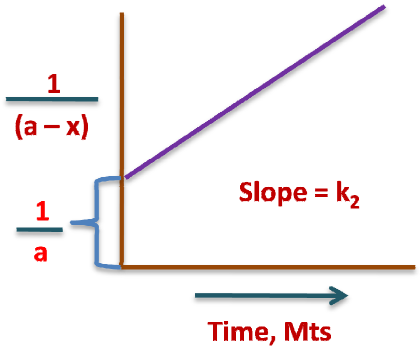
Calculate rate constant at each instant of time. And enter the values in the table given below.

Graphs:

i. Draw the graph ——— Vs. t A straight line graph passing through origin is obtained. The slope is equal to k_2.



ii. Draw the graph ——— Vs. t A straight line graph having intercept 1/a is obtained. The slope is equal to k_2.



Results:

Since the rate constant was found to be constant at different intervals of time it is 2nd order reaction.

i.e. order of reaction is 2.

Systematic Qualitative Analysis

Experiment	Observation	Interference
I. Preliminary tests		
1. Physical examination		
(a) Colour	Blue or bluish green Greenish Dark brown Pink Light pink or flesh colour White	Cu^{+2}, Ni^{+2} Ni^{+2}; Fe^{+2} Fe^{+3} Co^{+2} Mn^{+2} Shows the absence of Cu^{+2}, Ni^{+2}, Fe^{+3}, Mn^{+2}, Co^{+2}
(b) Smell	Ammonia smell Vinegar smell Smell like that of rotten eggs	(Ammonium salts) CH_3COO^- (Acetates) S^{2-} (Sulphides)
(c) Solubility (order of testing with the solvents)	i. Cold water ii. Hot water iii. Cold dil. HCl iv. Hot dil. HCl	**When the salt is soluble in a certain solvent, subsequent solvents are not taken to test the solubility**

2. Dry test Take a small quantity of the salt in a dry hard glass test tube and heat it gently first and then strongly	(a) Colourless and odourless gas which turns lime water milky (b) Colourless gas with pungent smell (c) Colourless gas with rotten eggs smell (d) Colourless gas with smell of ammonia (e) Reddish brown gas with pungent odour	May be Carbonate ion $[CO_3^{2-}]$ or a bicarbonate May be a Sulphite $[SO_3^{2-}]$ May be a Sulphide $[S^{2-}]$ May be ammonium salt $[NH_4^+]$ May be Nitrate $[NO_3^-]$ Nitrite $[NO_2^-]$ Bromide $[Br^-]$
3. Flame test Prepare a paste of the salt with a few drops of conc. HCl on a watch glass and place a small amount of this paste on glass rod and introduce it (oxidizing flame) into the flame	(a) Brick red (b) Crimson red (c) Apple green (d) Bright – bluish green (e) Green flashes	May be Calcium (Ca^{+2}) May be Strontium (Sr^{+2}) May be Barium (Ba^{+2}) May be Copper (Cu^{+2}) May be Zinc(Zn^{+2}) or Manganese(Mn^{+2})
4. Borax bead test (This is performed only for coloured salts) Borax is heated in the loop of platinum wire and is touched with small amount of coloured salt and is heated again in oxidizing flame of bunsen burner	(a) Green when hot, light blue when cold (b) Yellowish brown when hot, pale yellow when cold (c) Pinkish violet in both hot and cold (d) Brown when hot, pale brown when cold (e) Deep blue in both hot and cold	May be Copper (Cu^{+2}) May be Ferric $[Fe^{+3}]$ May be Manganese (Mn^{+2}) May be Nickel (Ni^{+2}) May be Cobalt $[Co^{+2}]$

5. Test for ammonium ion Take a pinch of salt in a test tube and add 1 – 2 ml of NaOH solution to it and heat. Bring a glass rod dipped in conc. HCl near the mouth of the test tube	White dense fumes are observed	May be Ammonium salt $[NH_4^+]$
6. Confirmatory test for $[NH_4^+]$ ion Take a pinch of salt in a test tube containing distilled water. Dissolve the salt. Add Nesseler's reagent to it	A chacolate brown coloured precipitate is observed	Confirms Ammonium ion $[NH_4^+]$

II. Identification of Acid radicals by wet tests

Preparation of sodium carbonate extract:

This is prepared only for those salts which are insoluble water.

A pinch of salt is mixed with 2 – 3 times the amount of sodium carbonate and is boiled with distilled water for some time. The suspension obtained is filtered. The filterate is known as sodium carbonate extract. (SCE). This is used in confirmatory tests of radicals

Division of acid radicals into groups

This part is classified into three parts

Group	Group reagent	Acid radicals
First group acid radicals	dil. HCl	Carbonate $[CO_3^{2-}]$ Sulphide (S^{2-}) Sulphite $[SO_3^{2-}]$ Nitrite $[NO_3^-]$

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Second group acid radicals	Conc. H_2SO_4	Chloride (Cl^-) Bromide (Br^-) Iodide (I^-) Nitrate $[NO_3^-]$ Acetate ($CH3COO^-$)
Third group acid radicals	dil. HCl + $BaCl_2$ solution dil. HNO_3 + Ammonium Molybdate	Sulphate $[SO_4^{2-}]$ Phosphate $[PO_4^{3-}]$

Experiment	Observation	Interference
I Group **1. Test with dil. HCl** Take a small quantity of the salt in a test tube and add 1 – 2 ml of dil. HCl	**(a) If colourless, odourless gas turns lime water milky is observed.**	May be Carbonation ion $[CO_3^{2-}]$
(a) Confirmatory test for carbonate radical Dissolve the salt in H_2O and add $MgSO_4$ solution	A white ppt is formed	Carbonate is confirmed
	(b) If colourless gas with suffocating smell is observed	May be Sulphite $[SO_3^{2-}]$
(b) Confirmatory test for sulphite To a portion of sodium carbonate extract add potassium dichromate solution acidified with dil. H_2SO	A green colour is obtained	Sulphite is confirmed

	(c) If colourless gas with rotten egg smell is observed	May be Sulphide ion (S^{2-})
(c) Confirmatory test for sulphide Sodium carbonate extract acidified with dil. acetic acid and add lead acetate solution	A black ppt is obtained	Confirms the presence of Sulphide
	(d) If reddish brown gas turns starch – iodide paper blue	May be Nitrite $[NO_2^{2-}]$
(d) Confirmatory test for nitrite Sodium carbonate extract is acidified with dil. acetic acid; Add a few drops of potassium iodide solution and a few drops of starch solution	Blue colouration is observed	Confirms the presence of Nitrite radical
II group **2. Test with Conc. H_2SO_4** Take a pinch of salt with conc. Sulphuric acid in a test tube. Identify the gas evolved in cold and then heat	**(a) Colourless gas with a pungent smell and gives white fumes when a glass rod dipped in ammonium hydroxide (NH_4OH) is exposed**	May be Chloride ion (Cl^-)
(a) Confirmatory test for chloride ion Acidify the sodium carbonate extract with dil. HNO_3, boil for some time, cool and add $AgNO_3$ solution	A white ppt is formed which is soluble in Ammonium hydroxide	Confirms the presence of Chloride radical
	(b) Reddish brown vapours with pungent smell,	May be Bromide ion (Br^-)

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	turns starch paper yellow	
(b) Confirmatory test for bromide Acidify sodium carbonate extract with dil. HNO_3. Boil, cool and add $AgNO_3$ solution	A light yellow ppt is obtained which is soluble in NH_4OH on adding excess and shaking	Confirms the presence of Bromide radical
	(c) Violet vapours, which turn starch paper blue and a layer of violet sublimate is formed on the sides of the test tube	May be Iodine ion (I-)
(c) Confirmatory test for iodide Acidify sodium carbonate extract with dil. HNO_3. Boil, cool and add $AgNO_3$ solution	A yellow ppt is formed which is insoluble in NH_4OH	Confirms the presence of Iodide
	(d) If brown fumes evolve, add Cu turnings to the reaction anixture. The solution acquires blue colour	May be Nitrate radical $[NO_3^-]$
(d) Confirmation of nitrate Acidify sodium carbonate extract with dil. H_2SO_4 and add a small quantity of freshly prepared ferrous sulphate solution. Pour conc. H_2SO_4 slowly along the walls of the test tube	A dark brown ring is formed at the junction of the layers of the acid and the solution	Confirms the presence of Nitrate radical
	(e) Colourless vapours with vinegar smell, which turns blue litmus red	May be acetate radical (CH_3COO^-)

(e) Confirmatory test for acetate Take water extract of the salt. Add neutral ferric chloride solution filter. Divide the solution into two portions	Reddish coloured filtrate	Confirms the presence of acetate ion
(i) To one part, add dil. HCl	Reddish colour disappears	Confirms the presence of acetate radical
(ii) To second part, add water and boil	Reddish brown ppt	Confirms the presence of acetate radical
3. Test for sulphate. Dissolve a pinch of salt in dilute HCl in a test tube and add few drops of $BaCl_2$ solution	A white ppt insoluble in conc. HCl is obtained	Confirms sulphate radical $[SO_4^{2-}]$
(a) Confirmation for sulphate To a part of the aqueous solution of the salt add barium chloride solution	A white ppt is formed which is insoluble in dil. HCl	Confirms the presence of sulphate radical
4. Test for phosphate with Ammonium Molybdate solution Add conc. HNO_3 to the given salt, boil and add excess of Ammonium Molybdate solution	A canary yellow precipitate is formed	May be phosphate radical $[PO_4^{3-}]$
(a) Confirmation of phosphate		

To the aqueous solution or sodium carbonate extract, add concentrated nitric acid and boil. Add Ammonium Molybdate solution in excess and again boil	A deep yellow ppt or colouration is observed	Confirms the presence of phosphate radical

III. Wet Tests for Identification of Cations

The cations indicated by the preliminary tests given above are confirmed by systematic analysis given below.

The first essential step is to prepare a clear and transparent solution of the salt. This is called original solution. It is prepared as follows.

Preparation of Original Salt Solution (O.S.):

To prepare the original solution, following steps are followed one after the other in a systematic order. In case the salt does not dissolve in a particular solvent even on heating, try the next solvent.

The following solvents are tried:

1. Take a little amount of the salt in a clean boiling tube and add a few mL of distilled water and shake it. If the salt does not dissolve, heat the content of the boiling tube till the salt completely dissolves.
2. If the salt is insoluble in water, take fresh salt in a clean boiling tube and add a few mL of dil.HCl to it. If the salt is insoluble in cold, heat the boiling tube till the salt is completely dissolved.
3. If the salt does not dissolve either in water or in dilute HCl even on heating, try to dissolve it in a few mL of conc. HCl by heating.

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Group Analysis:

Division of cations into groups

Group	Group reagent	Cations
Group I	Dilute HCl	Pb^{2+}
Group II	H_2S in the presence of dilute HCl	Cu^{2+}
Group III	NH_4OH in the presence of NH_4Cl	Fe^{3+}, Al^{3+}
Group IV	H_2S in the presence of NH_4OH	Ni^{2+}, Mn^{2+}, Zn^{2+}, Co^{2+}
Group V	$(NH_4)_2\,CO_3$ in the presence of NH_4OH	Ca^{2+}, Ba^{2+}, Sr^{2+}
Group VI	No	Mg^{2+}

For the analysis of cations belonging to groups I – VI, the cations are precipitated from the original solution by using the group reagents.

The separation of all the six groups is represented as below.

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Original solution

Dil. HCl

Precipitate, Group I
(Pb^{2+}) as chloride

If no precipitate is formed

Pass H_2S gas
into the solution

Precipitate, Group II
Cu^{2+} as sulphides

If no precipitate is formed. Take original solution

Heat (O.S.) with one drop of conc. HNO_3,cool and add solid NH_4Cl + NH_4OH solution in excess. i.e. till the smell of ammonia is observed after shaking the test tube

Precipitate, Group III
(Fe^{3+}, Al^{3+}) as hydroxides

If no precipitate is formed

Pass H_2S gas
into the solution

Precipitate, Group IV
(Co^{2+}, Ni^{2+}, Mn^{2+}, Zn^{2+}) as sulphides

If no precipitate is observed, take original solution

Add NH_4OH excess
and solid $(NH_4)_2Co_3$

Precipitate, Group V
(Ba^{2+}, Sr^{2+}, Ca^{2+}) as carbonates

If no precipitate is observed,
take original solution to test for Mg^{2+}

Experiment	Observation	Interference
1. Confirmatory test for Group – I cation (Pb^{+2}) Boil the precipitate with distilled water and divide the solution into two parts	**White precipitate – Pb^{+2}**	May be Pb^{+2}
(a) Part 1: Add potassium iodide to the first part	A yellow ppt is obtained	Confirms the presence of lead (Pb^{+2})
(b) Part 2: To the second part add	A yellow ppt is obtained which is soluble in NaOH	Confirms the presence of lead (Pb^{+2})

potassium chromate solution	solution and insoluble in ammonium acetate solution	
2. Confirmatory test for Group – II cation (Cu^{+2}) Boil the precipitate with distilled water and excess of Ammonium hydride solution	**Black precipitate – Cu^{+2}** A **blue solution** is obtained. Acidify the above deep blue solution with dil. acetic acid **Chocolate** brown ppt is obtained	May be Copper (Cu^{+2}) Confirms the presence of Copper (Cu^{+2})
3. Confirmatory test for Group – III cations	**Reddish brown ppt – Fe^{+3}**	May be Fe^{+3} radical
(a) Confirmation of Fe^{+3} Dissolve the brown ppt in dil. HCl and add potassium ferrocyanide solution	A prussion blue ppt/colouration appears	Confirms the presence of Fe^{+3}
(b) Confirmation of Al^{+3} Dissolve the white ppt in dil. HCl and add sodium hydroxide solution and warm	**White precipitate – Al^{+3}** A white gelatin on precipitates soluble in excess of sodium hydroxide solution	May be Al^{+3} radical Confirms the presence of Al^{+3}
4. Confirmatory test for group – IV cations	**Dirty white ppt – Zn^{+2}**	May be Zn^{+2} radical
(a) Confirmation for zinc Dissolve the white ppt in dil. HCl and add NaOH solution	White ppt is observed, which is soluble in excess of NaOH	Confirms the presence of Zinc (Zn^{+2})

(b) Confirmation for Manganese Dissolve the ppt in dil. HCl by boiling, then add sodium hydroxide solution in excess	**Flesh coloured precipitate – Mn^{+2}** A white ppt is formed which turns brown on keeping	May be Mn^{+2} radical Confirms the presence of Manganese (Mn^{+2})
(c) Confirmation for cobalt and nickel Note the colour of the salt.	**Black precipitate – (Ni^{+2} and CO^{+2})** If the salt is green If the salt is purple	May be Co^{+2} or Ni^{+2} Ni^{+2} radical may be present Co^{+2} radical may be present
i. Confirmation for nickel Dissolve the salt in water and make it alkaline by adding excess of NH_4OH and then add a few drops of dimethyl glyoxime	Formation of bright red ppt is observed	Confirms the presence of Ni^{+2} ions
ii. Confirmation for cobalt Dissolve the salt in water and add NH_4OH solution. Acidify it with dilute acetic acid and add a pinch of potassium nitrite	A yellow precipitate is observed	Confirms the presence of Co^{+2} ions
5. Confirmatory test for Group – V cations Dissolve a white ppt in dil. acetic acid. Boil off CO_2. The solution is divided into 3 parts		
(a) Confirmatory test for barium radical (i) To one part of the	Yellow precipitate is	Confirms the presence of

above solution, add $K_2Cr_2O_7$ solution.	obtained	Barium radical
(ii) Perform the flame test with the precipitate with $(NH_4)_2Co_3$ solution	Apple green flame is observed	Confirms the presence of Barium radical
(b) Confirmatory test for stroncium		
(i) To second part of the above solution, add ammonium sulphate	White precipitate is obtained	Confirms the presence of strontium radical
(ii) Perform the flame test with the precipitate obtained with $(NH_4)_2Co_3$	Crimson red flame is observed	Confirms the presence of strontium radical
(c) Confirmatory test for calcium		
(i) To the third part of the above solution add ammonium oxalate solution	A white ppt is obtained	Confirms the presence of Calcium radical
(ii) Perform the flame test with the precipitate obtained with $(NH_4)_2Co_3$	Brick red flame is observed	Confirms the presence of Calcium radical
6. Confirmatory test for Group – VI cations		
To the original solution add dil. HCl and treated with 1 drop of	A blue ppt is observed	Confirms the presence of Magnesium radical

magneson reagent and then add 2 – 3 ml of sodium hydroxide solution		

Organic analysis

Functional group tests:

Test	Observation	Result
Preliminary tests: 1. State 2. Colour 3. Odour 4. <u>Flame test</u> : Little amount of organic compound is taken in a spatula over a low flame.	Non-sooty flame Sooty flame	Aliphatic compound Aromatic compound
5. <u>Solubility</u> : In water	soluble	May be phenols, amines, aliphatic alcohols,carboxylic acids, aldehydes, ketones, carbo hydrates.
In ether	soluble	May be phenols, amines, aliphatic alcohols,aldehydes, ketones.

	(if water soluble, ether insoluble)	May be carbohydrates
In NaHCO3 solution	Soluble	May be carboxylic acids
In dil. NaOH solution	Soluble (soluble in NaHCO3)	May be phenols
In dil. HCl	Soluble(insoluble in NaHCO3)	Amines.
In conc. H2SO4 solution	soluble insoluble	May be alcohol, aldehyde, ketone. May be Aromatic compounds
Test for unsaturation: 0.1 (or) 0.2 ml of organic compound is dissolved in 2 ml of CCl_4 then 0.1 ml of bromic – water is added dropwise.	Brown colour of bromine discharged Brown colour of bromine is not discharged	Unsaturation is present No unsaturated is present.
Test for carboxylic group: 0.1 g (or) 0.2 ml of organic compound is taken in a test tube and added a pinch of $NaHCO_3$ to the organic compound	Briskeffervescence are formed No briskeffervescene are formed.	May be carboxylic group is present Carboxylic group is absent.

CHEMISTRY PRACTICALS LIST AND PROCEDURE

Conformation test: 0.1 g (or) 0.2 ml of organic compound is taken in a test tube and added 1 ml of ethanol (or) methanol and then 2 – 3 drops of con. H_2SO_4 is added the reaction mixture is heated for 10 – 15 min in a hot water bath at about 50^0 c. finally the reaction mixture is paired into a beaker containing aq. Na_2CO_3 Solution.	Sweet small of the substance is observed.	Carboxylic functional group is present.
Test for Phenolic group: 0.1 g (or) 0.2 ml of organic compound is taken in a test tube; to this 2 – 3 ml of neutral FeCl3 solution is added.	Green (or) violet colour is obtained Green (or) violet colour is not obtained.	May be phenolic group is present Phenolic group is absent
Conformation test: 0.1 g (or) 0.2 ml of organic compound is taken in a test tube to this 0.1 g of phthalic anhydride and 1 – 2 drops of con. H_2SO_4 is added. The test tube is heated about 1 min and then the reaction mixture is poured into the beaker containing 15 ml of dil. NaOH solution.	Pink (or) blue (or) green (or) red colour is appeared.	Phenolic group is present
Test for alcoholic group: A small piece of sodium is		

taken in a test tube to this 2 ml (or) 0.1 g of the given organic compound is added.	Effervescence are formed No effervescence are formed	May be alcoholic group is present. Alcoholic group is absent
Conformation test: 0.2 ml (or) 0.1 g of organic compound is taken in a clean dry test tube, to this 10 ml of 10% aqueous KI solution and 10 ml of freshly prepared NaOCl solution is added and warned gently.	Yellow crystals of iodoform separated	Alcoholic group is present
Test for carbonyl group: 0.2 ml (or) 0.1 g of organic compound is taken in a test tube and to thin 2 – 3 ml of 2, 3 di nitro phenyl hydrazine is added	Orange – yellow precipitate is formed Orange – yellow precipitate is not formed	Carbonyl group is present. May be aldehdye (or) ketone. Carbonyl group is absent
Conformation test: 0.1 g (or) 0.2 ml organic compound is taken in a dry test tube, to this 1 ml of freshly prepared Tollen's reagent is added. The reaction mixture is placed on water bath for 2 min.	Silver mirror is formed on inner side of test tube. Silver mirror is not formed.	Adehdyes is present Ketone is present
Test for carbohydrate (or) simple sugar: 0.2 ml (or) 0.1 g of aqueous solution of organic compound is taken in a clean dry test tube, to this, alcoholic α – napthol is	Violet colour ring is formed between two layers. Violet colour ring is not formed between two layers.	May be carbohydrate is present May be carbohydrate is absent

added. To this 2 – 3 ml of con. H_2SO_4 is added slowly along the sides of the test tube. The test is kwown as Molisch's test.		
Conformation test: 0.2 ml (or) 0.1 g of aqueous solution of organic compound is taken in a test tube to this 1 – 2 ml of benedict solution is added.	Red colour precipitate is formed	Carbohydrate group is present
Test for amines: 0.2 ml (or) 0.1 g of organic compound is taken in a test tube, to this few drops of dil. HCl is added.	Organic compound is dissolved.	May be primary amine is present.
Conformation test: 0.2 ml con 0.1 g of organic compound is taken in a clean dry test tube, to this 1 ml of con. HCl and few drops of CHCl3 is added, and then 2 ml of alc. KOH solution is added and warmed, for some time.	Offensive (or) obnoxious smell of carbylamines is evolved.	Primary amine is present